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Indian Standard

SPECIFICATION FOR
1, 4-DIAMINOANTHRAQUINONE, TECHNICAL

(First Revision)

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AMENDMENT NO. 1 APRIL 1994
TO
IS 6266 : 1983 1, 4-DIAMINOANTHRAQUINONE, TECHNICAL

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(*Page* 8, *clause* **A-4.1.3.1**, *line* 8) – Substitute '0.003' *for* '0.03'

(PCD 11)

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SPECIFICATION FOR 1,4-DIAMINOANTHRAQUINONE, TECHNICAL

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SPECIFICATION FOR 1,4-DIAMINOANTHRAQUINONE, TECHNICAL

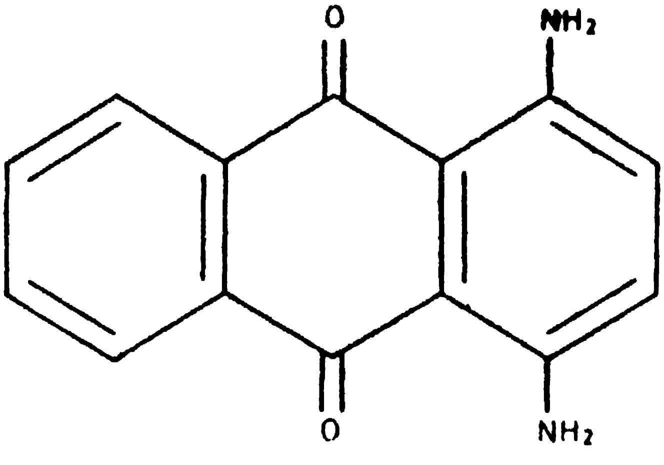
(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 10 February 1983, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was published in 1971. Taking cognizance of experience in the manufacture of this dye intermediate, the Sectional Committee decided to revise and up grade the standard. The value for assay of 85·0 percent has been increased to 88·0 percent. The requirement for residue insoluble in glacial acetic acid has been reduced from 8·0 to 5·0 percent. An additional requirement for melting range has been included which is a good indication of purity of the material.

0.3 1,4-diaminoanthraquinone ($C_{14}H_{10}O_2N_2$) is a very important intermediate in the manufacture of vat and disperse dyes. It is represented by the following structural formula:



1,4-Diaminoanthraquinone
(Molecular Mass 238)

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for 1,4-diaminoanthraquinone, technical.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of reddish-brown powder and shall be free from visible impurities.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR 1,4-DIAMINOANTHRAQUINONE, TECHNICAL

SL No	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL No. IN)	
			Appendix A	IS : 5299-1969*
(1)	(2)	(3)	(4)	(5)
i)	Moisture percent by mass, *Max*	0·5	—	9·3
ii)	Sulphated ash, percent by mass, *Max*	2·5	—	11·2
iii)	Hydroxyaminoanthraquinone and quinizarine content, percent by mass, *Max*	5·0	A-2	—
iv)	Residue insoluble in glacial acetic acid, percent by mass *Max*	5·0	A-3	—
v)	Assay percent by mass, *Min*	88·0	A-4	—
vi)	Melting range, °C	260 to 263	—	8

*Methods of sampling and tests for dye intermediates.

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*Rules for rounding off numerical values (*revised*).

3. PACKING AND MARKING

3.1 Packing — The material shall be suitably packed in polythene bags or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

3.2 Marking — Each container shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer and his recognized trade mark, if any;

c) Lot or batch number; and

d) Tare, net and gross mass.

3.2.1 The product may also be marked with Standard mark.

3.2.2 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in **3** of IS : 5299-1969*.

4.2 Number of Tests — Tests for the determination of all the characteristics specified in this standard shall be conducted on the composite sample.

4.3 Criteria for Conformity — For declaring the conformity of the lot to this standard, the test results for each of the characteristics shall satisfy the relevant requirement specified in this standard.

5. METHOD OF TEST

5.1 Tests shall be carried out according to the methods prescribed in Appendix A and IS : 5299-1969*. Reference to relevant clauses are given in col **4** and **5** of Table 1.

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*Methods of sampling and tests for dye intermediates.

APPENDIX A

(*Table* 1 and *Clause* 5.1)

METHODS OF TEST FOR 1,4-DIAMINOANTHRAQUINONE, TECHNICAL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF HYDROXYAMINOANTHRAQUINONE AND QUINIZARINE CONTENT (OXYAMINO DERIVATIVES)

A-2.0 Outline of the Method — The material is dissolved in pyridine and then alkali soluble matter is separated by distilling off the pyridine. It is acidified, filtered through a sintered-glass crucible, dried and weighed.

A-2.1 Procedure — To determine the oxyamino derivatives (alkali solubles), weigh accurately about 5 g of the sample (dried and sieved) in a 500-ml flask, and add 100 ml of pure pyridine. Gradually heat to dissolve this sample completely. Five grams of potassium hydroxide, technical (88 to 90 percent pure), is separately dissolved in 250 ml distilled water and this solution is slowly added to the solution of the sample made with pyridine. Pyridine is then steam distilled out of this mixture till the distillate shows a neutral reaction with brilliant-yellow paper. The residue is then filtered by suction over a buchner funnel of 150 mm diameter, using a filter paper, and washed with 1 percent potassium hydroxide solution till the filtrate is colourless. The filtrate, together with the washings, is then acidified with dilute hydrochloric acid (1 : 1, *v/v*), boiled and kept as such overnight. Next day, the precipitate is filtered out over a tared sintered-glass crucible of porosity G4 and washed acid free. It is then dried at 100°C and cooled in a desiccator before weighing. Repeat heating and cooling till a constant mass is obtained.

A-2.2 Calculation

$$\text{Hydroxyaminoanthraquinone and quinizarine content, percent by mass} = \frac{M_1}{M_2} \times 100$$

where

M_1 = mass in g of the residue, and

M_2 = mass in g of the sample taken for test.

---

*Specification for water for general laboratory use (*second revision*).

A-3. DETERMINATION OF RESIDUE INSOLUBLE IN GLACIAL ACETIC ACID

A-3.1 Procedure — Weigh accurately about 5 g of the dried and sieved sample and place it in a 500-ml flask, along with 300 ml of glacial acetic acid. The mixture is boiled for one hour under reflux and then filtered hot over a tared sintered-glass crucible of porosity G1. The residue on the crucible is washed with hot acetic acid until the filtrate is colourless. The washing is continued till the residue is acid free, which is then dried at 100°C, and weighed after cooling in a desiccator. Heating and cooling is continued till the last two weighings give a constant mass.

A-3.2 Calculation

$$\text{Residue insoluble in glacial acetic acid, percent by mass} = \frac{M_3}{M_1} \times 100$$

where

M_3 = mass in g of the residue, and

M_1 = mass in g of the sample taken for test.

A-4. ASSAY (BY CHROMATOGRAPHY)

A-4.1 Reagents

A-4.1.1 *Alumina* — Neutral. Column chromatograhic grade.

A-4.1.1.1 *Deactivation of standard alumina* — 100 g of standard alumina is deactivated by adding 3 ml of distilled water and mixing thoroughly by shaking vigorously for 15 minutes.

A-4.1.2 *Toluene* — Dried for 24 hours over fused calcium chloride, filtered and distilled. Portion distilling between 109 and 111°C is collected for use.

A-4.1.3 *1,4-Diaminoanthraquinone* (*Chromatographic Grade*) — Dissolve 0·2 g of 1, 4-diaminoanthraquinone, technical, in 300 ml of toluene and reflux for 5 to 10 minutes. Cool the solution to room temperature. Transfer the solution gradually to the chromatographic column prepared with deactivated alumina and dry toluene as described in **A-4.2.1**. Allow the solution to pass through the column and elute further with toluene. Reject the coloured bands of impurities, namely, light orange and blue, and collect the first violet band (the main band) of 1, 4-diaminoanthraquinone. A battery of 5 to 6 such tubes shall be kept in operation for collecting a sizeable quantity of pure material. Filter the collected solution through a filter paper to remove any extraneous matter. Concentrate the solution, cool and allow to crystallize. Filter off the crystallized 1, 4-diaminoanthraquinone. Cool and filter on a sintered-glass crucible of porosity G4. Wash and dry to constant mass in a vacuum oven at 100°C.

A-4.1.3.1 In order to check its purity, dissolve about 0·01 g (accurately weighed) in 10 ml of toluene and pass the cooled solution through a chromatographic column, set up as described in **A-4.2.1**, eluting with toluene, as necessary. Collect the band carefully (only one band should appear) and determine its optical density at 545 nm. Make up a solution of purified crystals using the same proportion of crystals to solvent. Determine its optical density in the same way. A difference in the two figures for optical density, amounting to more than 0·03 indicates the presence of an impurity. The chromatographic purification shall then be repeated until this check test is satisfied.

A-4.2 Apparatus

A.4.2.1 *Chromatographic Column*

A-4.2.1.1 A glass tube 40 cm long and having 1·5 cm internal diameter, joined with a 50-ml thistle funnel at the upper end and fitted with a stop-cock at the lower end, is set up vertically, such that the percolation passing through the tube can be collected conveniently in a 250-ml conical flask. Place a cotton-wool plug in the tube and press it to the bottom of the tube by means of a glass rod, flattened at the end. Place a disc of filter paper cut to the approximate internal diameter of the chromatographic tube on top of the cotton wool.

A-4.2.1.2 Prepare a slurry of about 15 g of the deactivated alumina in toluene and pour it into the tube. Wash down the sides of the tube and pack the column by lightly tapping the tube. Place first a disc of filter paper and then a cotton wool plug at the top of the alumina column. Care should be taken to see that the alumina column remains soaked with the solvent, such that at the least a 2 cm layer of the solvent always remains on top of the alumina. On no account shall the alumina be left to dry. In the event of this happening, the alumina in the tube shall be re-slurried and re-packed.

A-4.3 Standard Calibration Graph — Prepare a number of standard solution of chromatographically pure 1, 4-diaminoanthraquinone, as obtained in **A-4.1.3** in toluene, varying in concentrations from 0·6 mg/100 ml to 2·0 mg/100 ml, with a difference of 0·2 mg/100 ml between successive concentrations. Take readings for optical density or percentage transmittance for the concentrations at a wavelength of 545 nm (*see* Notes) using the specially matched cells specified for the particular spectrophotometer used. The temperature of the solutions immediately before and after measurement shall be 27 ± 2°C. Where the instrument is calibrated in percentage transmittance only, the optical density can be read from standard tables. Plot the calibration curve of concentration (in mg/100 ml) against optical density. This is the standard calibration graph.

NOTE 1 — The readings for optical density or percentage transmittance are taken at the predetermined wavelength at which there is maximum absorption.

NOTE 2 — The standard calibration curve and the spectrophotometer shall be checked for accuracy from time to time.

A-4.4 Procedure

A-4.4.1 Weigh accurately 100 to 120 mg of the sample in a 100-ml clean, dry conical flask. Add about 80 ml of toluene and reflux for 10 to 15 minutes. Cool and transfer the solution to a 100-ml volumetric flask. Wash the conical flask with toluene and then fully transfer the material to the volumetric flask. Dilute the solution to the 100-ml mark. Pipette out 25 ml from this solution and transfer to another 100-ml volumetric flask and further dilute to the 100-ml mark with toluene. With the help of a 10-ml burette, transfer 10 ml of this latter diluted solution accurately to the chromatographic column (prepared as in **A-4.2.1**). The solution starts passing down the alumina column. When it is just fully absorbed at the top of the alumina, wash carefully with small quantities of toluene. Finally, fill the upper part of the column with toluene and allow the chromatogram to develop, keeping a good head of solvent above the alumina column throughout. Reject the coloured bands preceding the main band of 1, 4-diaminoanthraquinone which is violet in colour. Start collecting this elute, when the main band is about 1·5 cm from the lower tip of the column in a 250-ml volumetric flask. When the entire band of 1, 4-diaminoanthraquinone is thus eluted, dilute to 250 ml exactly with toluene (solution A). Use this solution to take readings from the optical instrument.

A-4.4.2 Adjust the wavelength of maximum absorption (λ max) at 545 nm, and then adjust the instrument to 100 percent transmittance with toluene blank. Now replace the blank with solution (*A*) of the sample and read the percentage transmittance. Refer to the standard tables for conversion of percent transmittance to optical density. Read out the concentration against the optical density from the standard calibration graph (*see* **A-4.3**) in terms of milligrams per 100 ml of the final dilute soution (solution *A*).

A-4.5 Calculation

A-4.5.1 The amount of 1, 4-diaminoanthraquinone in the sample is $C \times 100$ mg.

Thus

$$\text{Assay, percent by mass} = \frac{10\,000\,C}{M_1}$$

where

C = quantity of 1, 4-diaminoanthraquinone in milligrams contained in 100 ml of the final dilute solution (solution A) as read from the calibration graph, and

M_1 = mass in mg of the sample taken for test.

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